

An electrochemical impedance spectroscopy and scanning electron microscopy study of the influence of positive plate compression on the electrochemical behaviour of lead-acid batteries

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Abstract

This study has developed an electrochemical impedance spectroscopy (EIS) method for the in situ investigation of the influence of positive plate compression on the electrochemical behaviour of lead-acid batteries during charge/discharge cycling. The EIS data for a fully charged and fully discharged battery are internally consistent with the expected kinetics of a battery in the opposite states of charge, and demonstrate that EIS measurements may be recorded with a high level of reproducibility. Furthermore, this study has necessitated the development of a special cell incorporating horizontally orientated battery plates that can be subjected to elevated pressure through the stacking of lead bricks on top of the cell, as well as a physically robust reference electrode system that can withstand the application of pressure. For this purpose, a platinum-wire pseudo-reference electrode has been developed, and has been shown to exhibit sufficient electrode stability over the period of an EIS recording, enabling the measurement of reproducible and meaningful EIS data. Additionally, the influence of positive plate compression on the behaviour of the lead-acid battery has been investigated by using scanning electron microscopy (SEM). Clearly, the experimental data show that plate compression enhances significantly the kinetics and concomitant performance of the lead-acid battery, and this is related to the enhanced reactivity of the active material, as rationalized by using the agglomeration-of-spheres (AOS) model.

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1. Introduction

There is a significant body of previous research (e.g., references [1–9]) showing that the application of a small amount of mechanical pressure to the positive electrode enhances the efficacy of the lead-acid battery. The present research has investigated systematically the chemical and physical factors responsible for the enhancement of battery performance by positive electrode compression. This information may be used to enhance the energy output and

durability of maintenance-free lead-acid batteries to increase their competitiveness in renewable energy applications, such as remote area power supply (RAPS), solar power, diesel, etc.

The seminal paper by Takahashi et al. [8] studied the physical changes in the positive active mass of lead-acid batteries during deep discharge cycling, and found that a compression pressure of 100 kg dm² was able to dramatically extend the cycle-life of batteries under deep discharge cycling. Takahashi et al. [8] also demonstrated that compression suppresses significantly any structural changes taking place in the positive active mass (PAM), and maintains the cohesion of the particles in the PAM.

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The present research is underpinned by the primary hypothesis that an enhanced knowledge of factors influencing the efficacy of maintenance-free lead-acid batteries, which have been subjected to positive electrode compression, may enable the development of longer life batteries. The data obtained in this study has been used to confirm the validity of existing models (Winsel et al. [7], Takahashi et al. [8] and Edwards and Schmitz [9]) rationalizing the origins of battery performance enhancement through positive electrode compression. This outcome might be useful to the manufacturers of lead-acid batteries (e.g., traction and automotive) for the development of high performance renewable energy battery systems (e.g., RAPS, solar, diesel, hybrid, etc.). This will aid significantly the world-wide imperative to reduce greenhouse gas emissions through the use of renewable energy battery systems.

Although the theoretical considerations of Winsel et al. [7], Takahashi et al. [8] and Edwards and Schmitz [9] have provided very useful treatments for the phenomenon of agglomeration-of-spheres (AOS) [7] that is responsible for the enhancement of battery performance under the conditions of plate compression, there is scope for a phenomenological study of the influence of plate compression on the electrode kinetics and physical attributes of positive active material, and their concomitant effects on the performance of lead-acid batteries, as has been undertaken in the present study.

Electrochemical impedance spectroscopy (EIS) is a powerful electrode kinetic technique that can provide useful information about the electrochemical reactivity of lead-acid batteries as a function of state of charge in conjunction with the influence of compression of the positive active material. Several authors [10–15] have demonstrated that EIS is capable of yielding important information about the influence of state of charge on the electrochemical reactivity of lead-acid batteries, and the present study has adapted EIS to a rigorous study of the influence of plate compression on the behaviour of batteries.

Moreover, the present research has addressed the following specific aims: (1) studied *in situ* the effects of electrode compression on the electrode kinetics of reaction processes in the positive electrode by using EIS; (2) investigated the effects of positive electrode compression on the chemical and physical properties (i.e., corrosion layer and active material) of positive plates by using SEM.

2. Experimental techniques

2.1. Preparation of pasted plates

Positive plates were prepared using the paste formula given in Table 1 (Manders et al. [16]). Barton-pot leady oxide obtained from the New South Wales plant of Exide Battery Technologies and Analytical Reagent Grade sulfuric acid (BDH, AnalaR) were used in the preparation of

Table 1
Paste formula for the preparation of positive plates

Component	Positive paste
Leady oxide (kg)	2
Fibre (g)	0.6
Carboxymethyl cellulose (g)	5
1.40 sp. gr. H ₂ SO ₄ (mL)	132
Water (mL)	300
Acid-to-oxide ratio (wt.%)	4.7
Paste density (g mL ⁻¹)	4.6–4.7

paste. The paste mixing procedure entailed the addition of five separate 50 mL quantities of ultra-high-purity water to the nominal quantity of dry ingredients (i.e., leady oxide, fibre and carboxymethyl cellulose) with mixing for 2 min after each addition. Acid was then added to the mixture (four separate additions of 33 mL), and the paste was mixed for 2 min after each addition. Finally, the remainder of the water was added to the paste, and its density was recorded after 2 min of mixing by measuring the volume of water displaced by approximately 100 g of paste. The paste was then applied by hand to lead–calcium–tin grids that are commonly used in the manufacture of Exide Powersafe batteries.

The cureless paste plates were subjected to pressing in order to dry and improve the integrity of the active material. The pressing of plates entailed the stacking of four lead bricks (each weighing 11 kg) on top of six plates that were individually wrapped in one absorptive glass mat separator and sandwiched between two metal plates. The battery plates were pressed for 45 min before replacing the glass mat separators with fresh ones, and pressing for another 45 min.

2.2. Assembly of 2 V cells

A range of cureless plates was assembled into 2 V cells comprising 1 positive plate surrounded by two negative plates. The cured negative plates were produced in the factory of Exide Battery Technologies using a paste recipe and curing conditions similar to those described elsewhere by Manders et al. [16]. The positive and negative plates were separated using absorptive glass-fibre separators (approximately 3 mm thick) manufactured by Exide Battery Technologies, and the cells were placed in Exide Powersafe battery containers. A pair of 0.6 mm diameter platinum wires was placed on either side of the positive plate (also separated with glass-fibre) to be used as pseudo-reference electrodes in EIS measurements, and Fig. 1 depicts the cell assembly. The plates were formed in 240 mL of 1.07 sp. gr. H₂SO₄ using a constant current of 1 A for 6 h, and then rested at open circuit for 1 h to allow the sulfuric acid depleted diffusion layers of the pores to be replenished by the permeation of fresh electrolyte. The charge/rest period was repeated, followed by a final charge at 1 A for 6 h. After formation, the sulfuric acid and separators were discarded, and the cells were reassem-

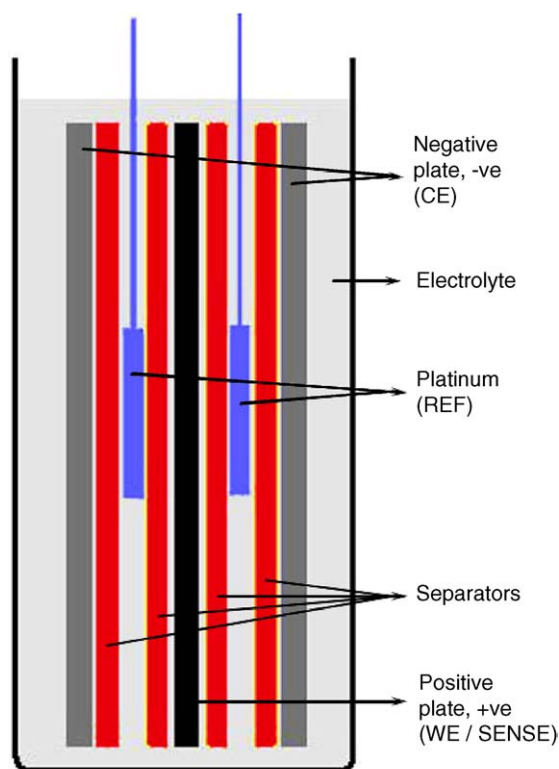


Fig. 1. Schematic diagram of the electrochemical cell used to conduct EIS measurements on lead-acid batteries. The negative plate was employed as the counter electrode (CE), while a platinum pseudo-reference electrode (REF) was employed so as to withstand the application of compressive loads, and the positive plate was made the working electrode (WE). Reprinted from A. Rochliadi, R. De Marco, A. Lowe, *J. Appl. Electrochem., Synergistic Effects of Novel Battery Manufacturing Processes for Lead-acid Batteries. Part II. Mechanistic Studies*, vol. 34, Page No. 269, Copyright (2004), with permission from Kluwer.

bled and filled with 1.270 sp. gr. H_2SO_4 . In most cases, an electrolyte-to-positive plate material ratio of 4.3 mL g^{-1} was used.

The cell was placed horizontally in a rigid plastic battery container and a rigid plastic compression plate was placed on top. The desired amount of pressure was exerted on the cell by loading the compression plate with a given mass of lead bricks. The plastic container was then filled up to the compression plate with the electrolyte. This arrangement allows the cell to expand and contract during cycling whilst maintaining a constant pressure. Note that all solder joints on the plates were insulated from the electrolyte by a PVC sleeve encapsulated within a moulded epoxy (Struers Epofix Kit) seal.

It is important to note that small sections of full-sized positive plates were employed in this study, so as to enable the application of the requisite pressures (i.e., $>100 \text{ kPa}$) by gravitational forces exerted by a stack of lead bricks. Accordingly, each positive plate had dimensions of $55 \text{ mm} \times 85 \text{ mm}$, and was approximately 18% of the surface area of a full-sized cell.

2.3. Charge/discharge cycling

The cycle-life performance of 2 V cells was assessed using a charge/discharge regime designed to accelerate battery failure by employing a Digatron BTS-500 computerized charge/discharge unit (Digatron GmbH, Germany) to undertake repetitive charge/discharge cycling at the 3-h rate. After charging to 2.55 V at 1 A, the cells were held at the top-of-charge voltage until the current had tapered to 0.18 A. At this juncture, the cells were discharged at 1 A until a voltage of 1.75 V was reached. The charge/discharge procedure was repeated until the discharge capacity fell to 60% of its original value.

2.4. Kinetic study by EIS

EIS studies were performed by using a Solartron SI 1287 as the electrochemical interface, and the Solartron SI 1260 as the impedance/gain-phase analyser. Negative plates were used as the counter electrode, and the positive plate was the working electrode. A separate pseudo-reference electrode of platinum was placed between the positive and negative plates. After some experimentation, the reference was chosen to consist of a Ag/AgCl electrode capacitively coupled ($100 \mu\text{F}$) to both of the platinum pseudo-references. The Ag/AgCl reference was placed in the bulk electrolyte solution. In fact, the Ag/AgCl reference was equilibrated in a separate batch of battery strength acid until its potential had reached a constant value, and transferred to the fresh battery electrolyte after stabilization. The constancy in reference electrode potential is consistent with an alteration in the AgCl surface to Ag_2SO_4 , removing all likelihood of a sluggish sulfate-based electrode reaction in battery electrolyte causing little or no drift in the reference sensor as a function of pressure, as well as contamination of the electrolyte by chloride. With this assembly, the working and counter electrodes set up a net current while two separate reference electrodes (sense/positive plate and REF/platinum pseudo-reference) are placed to measure the potential difference between the two points where the impedance is to be measured. By using separate cables for current and potential sensing, potential drops in the cabling are eliminated from the measurement.

A study of the stability of the platinum pseudo-reference electrodes in the 1.27 sp. gr. sulfuric acid electrolyte (not shown) demonstrated that these pseudo-reference electrodes acquire a high level of stability (viz., 0.01 mV min^{-1} drift) after approximately 4 h. As each EIS run takes about 15 min, this shows that the platinum electrodes are stable to 0.15 mV over the period of an EIS measurement, which is acceptable for EIS measurements.

EIS measurements were taken on each cell: (1) after formation; (2) at full charge at various stages of charge/discharge cycling; (3) at full discharge at various stages of charge/discharge cycling. Table 2 summarises the conditions used for the EIS measurements, noting that cycling was halted during the EIS measurements. Also noteworthy

Table 2
Experimental conditions for EIS measurements

Battery condition	EIS control	Amplitude	Bias	Frequency range
Formed	Current	60 mA	None	10 kHz to 10 mHz
Fully charged	Current	60 mA	5 mA ^a	10 kHz to 10 mHz
Fully discharged	Current	60 mA	None	10 kHz to 10 mHz

^a The 5 mA bias is used to ensure the cell remains in the fully charged state.

is the fact that the open cell potentials of the lead-acid battery cells were practicably constant during charge/discharge cycling (i.e., no meaningful trends were observed within the bounds of experimental uncertainty) immediately following full charging and full discharging whilst the EIS measurements were taken over a comparatively short-time frame compared to the length of a cycle.

2.5. Tear down analysis of failed cells

Prior to tear down analysis, the failed cells were returned to the same fully charged state by charging at 0.72 A up to 2.55 V, and the cells were held at this top-of-charge voltage until the current had tapered to 0.18 A. At this juncture, the failed cells were dismantled, and positive plates were rinsed in distilled water followed by drying at 50 °C for 24 h.

The grid corrosion layers of epoxy mounted, polished cross sections of positive plates were examined by SEM using a Phillips XL30 electron microscope operated at an accelerating voltage of 25 kV and a spot size setting of 4. The specimens were ground successively on 500, 1000 and 1200 grit emery paper using a water lubricant and a Struers Dap-V disc polisher that was rotated at 300 rpm. After grinding, the specimens were polished successively on Struers DP-Spray, P diamond sprays (3 and 1 μm) that had been applied to DP-Nap and DP-Mol felt pad discs using a rotation speed of 300 rpm and the Struers DP-lubricant Red.

3. Results and discussion

3.1. Charge/discharge cycling results

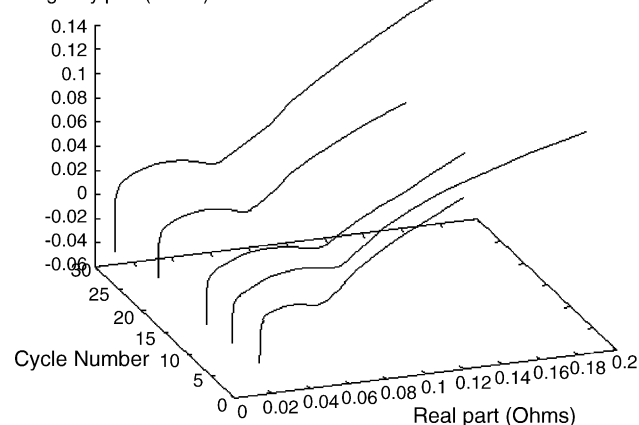
Charge/discharge cycling data for cells subjected to compression pressures of 0, 40 and 160 kPa (not shown) demonstrated a significant improvement in battery performance as a function of compression using an arbitrarily assigned point of failure when a cell's discharge capacity had fallen to 60% of its original value. At a compression pressure of 160 kPa, the cell did not fail, even after 500 repetitive charge/discharge cycles, while the uncompressed cell failed after 30 cycles, and the cell subjected to a compression pressure of 40 kPa failed after approximately 60 cycles. Notably, the average initial discharge capacities of these sub-sections of full-scale lead-acid battery positive plates (18% of a full-sized plate) were of the order of $86 \pm 8 \text{ Ah kg}^{-1}$, which is comparable to the active mass utilization of full-sized plates under identical formation, charging and discharging condi-

tions, as observed elsewhere by De Marco and co-workers [17,18].

3.2. EIS results

The complex-plane impedance plots, as a function of charge/discharge cycling, for cells subjected to 0, 40 and 160 kPa compression pressures are presented as 3D stacks in Figs. 2–4, respectively. In each case, an EIS run was performed in both the fully discharged (labelled as (a) in each of Figs. 2–4), as well as fully charged (labelled as (b) in each of Figs. 2–4) states.

(a) Electrochemical Impedance at Full Discharge - Uncompressed (f00a)
Imaginary part (Ohms)



(b) Electrochemical Impedance at Full Charge - Uncompressed (f00a)
Imaginary part (Ohms)

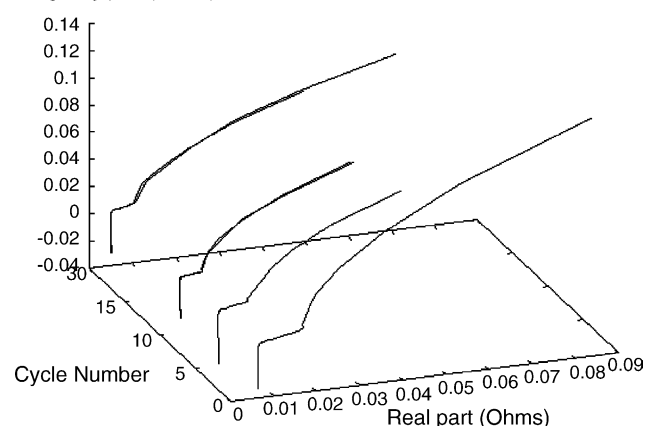
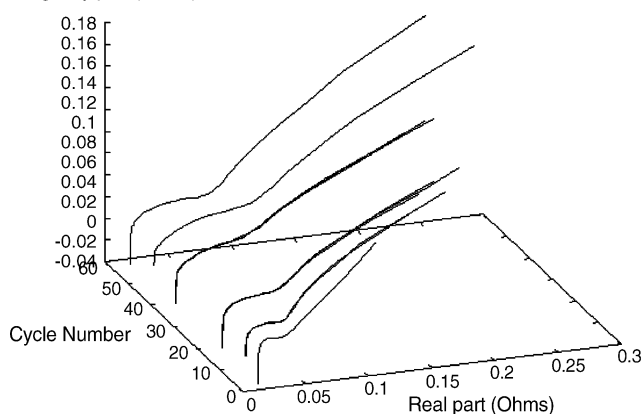


Fig. 2. A 3D stack of EIS complex-plane impedance plots as a function of charge/discharge cycling for an uncompressed cell in (a) the fully discharged and (b) the fully charged states.

(a) Electrochemical Impedance at Full Discharge -40 kPa Compression (f02a)
Imaginary part (Ohms)



(b) Electrochemical Impedance at Full Charge -40 kPa Compression (f02a)
Imaginary part (Ohms)

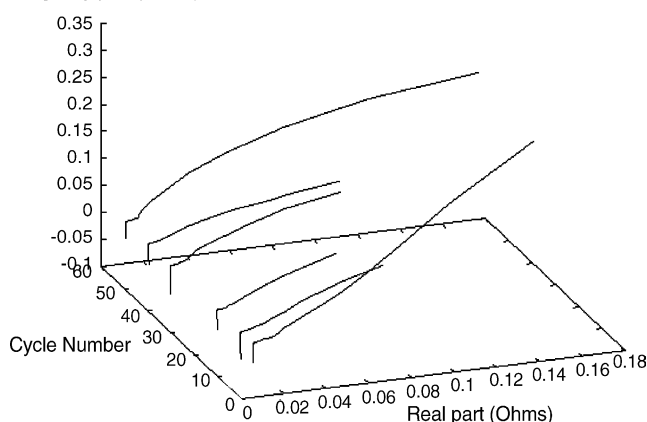
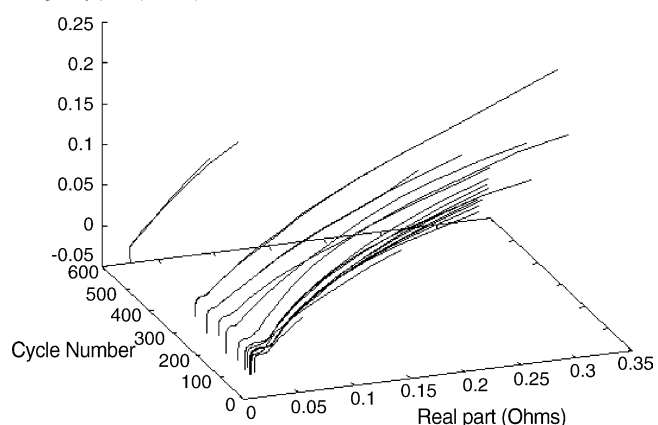


Fig. 3. A 3D stack of EIS complex-plane impedance plots as a function of charge/discharge cycling for cell compressed at 40 kPa in (a) the fully discharged and (b) the fully charged states.

It can be seen that there is little difference in the complex-plane impedance plots of the cycled cells, noting that all cells demonstrated a large phase angle at full charge, indicative of ideal capacitor behaviour. However, in the fully discharged state, the complex-plane impedance plots (see Figs. 2a, 3a and 4a) for both the uncompressed cell and the cell subjected to a compression pressure of 40 kPa revealed a 45° sloping line at low frequency, which is indicative of Warburg diffusion for the semi-infinite linear diffusion of electrolyte to the electrode surface. Nevertheless, the cell subjected to a compression pressure of 160 kPa did not display this Warburg behaviour, which indicates that the electrochemical processes are kinetically controlled, rather than diffusion controlled, at high compression pressures (viz., >160 kPa), probably due to the elevated diffusion rate of sulfuric acid into the finer pores of the active material at elevated compression pressures.

The equivalent electrical circuits best describing the EIS data are presented in Fig. 5a and b, and represent a slight modification of the circuit proposed by Pavlov and Petkova [12]. For the data reported in this study, the inductance element

(a) Electrochemical Impedance at Full Discharge -160 kPa Compression (f08)
Imaginary part (Ohms)



(b) Electrochemical Impedance at Full Charge -160 kPa Compression (f08)
Imaginary part (Ohms)

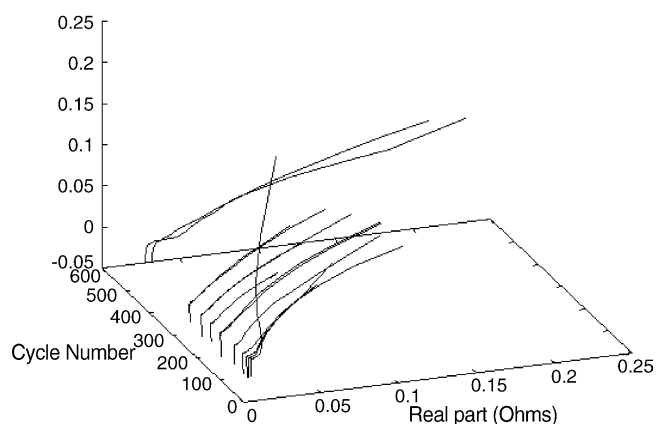


Fig. 4. A 3D stack of EIS complex-plane impedance plots as a function of charge/discharge cycling for cell compressed at 160 kPa in (a) the fully discharged and (b) the fully charged states.

(L1) was added to Pavlov's and Petkova's original circuit. Note that R1 gives the total ohmic resistance of the electrode, viz., the resistance of the active material (AM) of the positive plate, the resistance of the thin layer connecting the AM and the corrosion layer (CL), i.e., CL + AMCL along with the resistance of the electrolyte, L1 is the inductance caused by

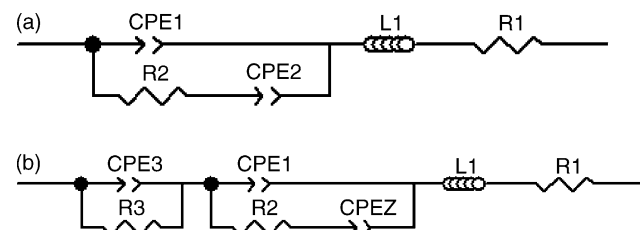


Fig. 5. EIS equivalent circuit models for (a) charged lead-acid batteries and (b) discharged lead-acid batteries. Reprinted from A. Rochliadi, R. De Marco, A. Lowe, *J. Appl. Electrochem.*, Synergistic Effects of Novel Battery Manufacturing Processes for Lead-acid Batteries. Part II. Mechanistic Studies, vol. 34, Page No. 269, Copyright (2004), with permission from Kluwer.

the metallic connection between the poles and electrodes of the battery, CPE1 is a constant phase element representing the dielectric properties of the reaction layers, which is equivalent to an electrical capacitance, CPE2 is a constant phase element representing the diffusion processes for H^+ and SO_4^{2-} ion in the reaction layer, and R_2 is the charge transfer resistance (R_{CT}) in the reaction layer, representing the electrochemical reaction process occurring at the interface and within the pores of the AM and CL + AMCL. During discharge, the electrochemical reaction shifted from the bulk of the AM to the CL + AMCL and necessitated the addition of new elements, R_3 and CPE3. Note that R_3 is the ohmic resistance of this CL + AMCL, and CPE3 is a constant-phase element representing the dielectric properties of the CL + AMCL layer [12].

Contrary to Pavlov's and Petkova's previous work [12], there were no significant trends in any of the equivalent circuit fitted parameters as a function of charge/discharge cycling of the cells. Nevertheless, the circuit fitted data provided a variance of approximately 20% relative for any type of cell, which is typical of the uncertainty associated with impedance measurements and equivalent circuit fitting, so the authors have employed average equivalent circuit fitted parameters for each type of cell to ascertain if there were any trends in the EIS parameters of the cells that were subjected to different compression pressures.

Significantly, there was a significant trend between the compression pressure and the mean charge transfer resistance for cells in both the fully discharged and fully charged states (refer to Fig. 6a and b). The discharged cells showed an increase in the charge transfer resistance at elevated compression pressures, and this behaviour is indicative of enhanced utilization of the AM in the positive plate at elevated pressures. In the case of the fully charged cells, however, this relationship is reversed, which fits the expected scenario of an enhanced efficiency of charging for compressed cells, which converts greater quantities of insulating lead sulfate into conductive AM, thereby lowering the charge transfer resistance. This evidence symbolises that the kinetics of the lead-acid battery electrochemistry is aided significantly by compression via the facilitated rate of reaction in the fully charged state, due to the AOS providing enhanced utilization of the AM in the positive plate on discharge, thereby increasing the amount of insulating lead sulfate and the concomitant charge transfer resistance for discharged cells as a function of compression pressure. Expectedly, the charge transfer resistance for the fully charged cell (mostly comprising conductive active materials) is significantly lower than that for the fully discharged cell (comprising mostly insulating lead sulfate).

The size of the initial semicircle, due to the reduction of lead dioxide at the CL + AMCL interface [12], shows a significant diminution at elevated compression pressures. This is confirmed by the concomitant decline in resistance obtained from the equivalent circuit fits with increased compression pressure (refer to Fig. 7a). At lower frequency, the 160 kPa

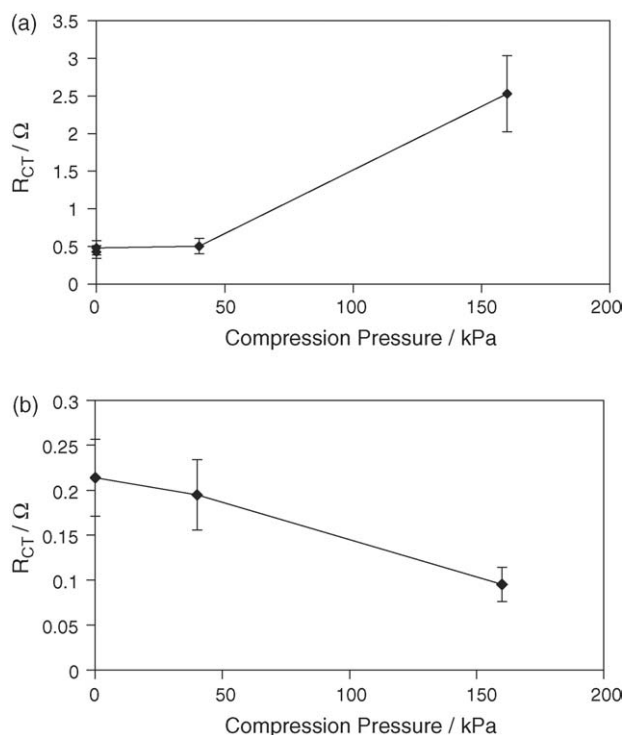


Fig. 6. Variation in the mean charge transfer resistance as a function of plate compression pressure for (a) fully discharged and (b) fully charged cells.

compressed cell tended towards a comparatively high capacitance against the other cells (see Fig. 7b). These results indicate a higher reaction layer capacitance and concomitant reaction rate [12] at the CL + AMCL interface as a function of compression pressure, and highlights that this region of the positive plate is a crucial one for regulating the efficacy of the lead-acid battery via plate compression.

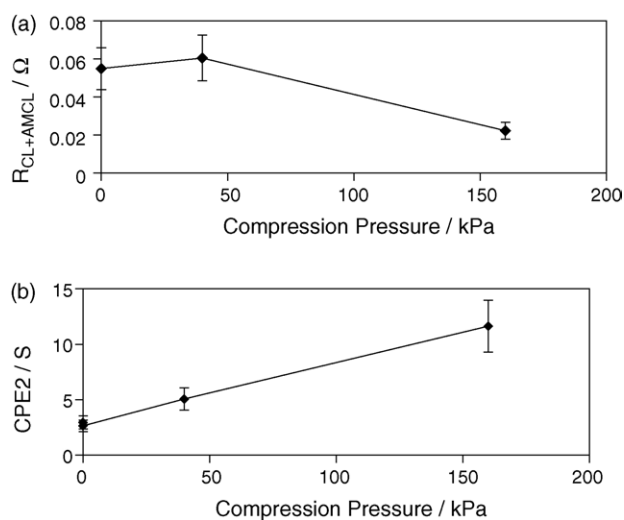


Fig. 7. Plots of the (a) mean ohmic resistance and (b) mean capacitance of CPE2 at the CL + AMCL interface for fully discharged cells as a function of the compression pressure.

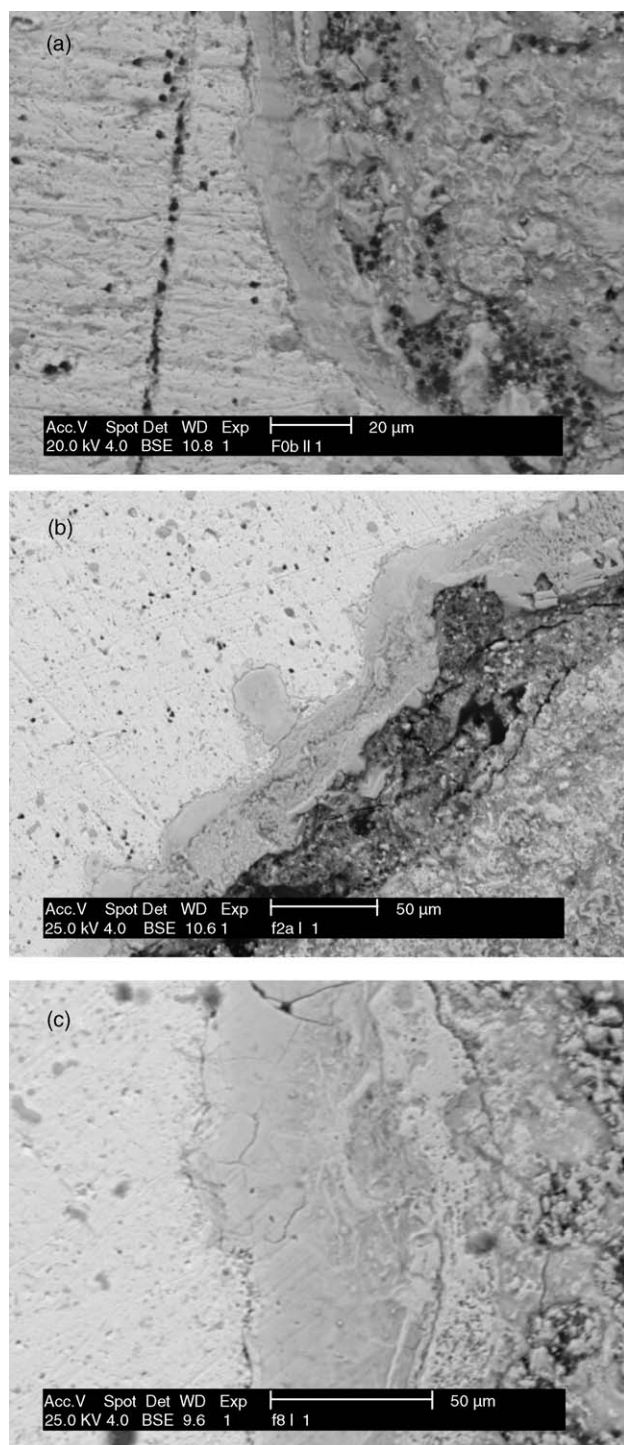


Fig. 8. SEM secondary electron images of polished cross-sections of cycled positive lead-acid battery plates: (a) uncompressed cell; (b) cell compressed at 40 kPa; (c) cell compressed at 160 kPa.

3.3. SEM characterization

A very interesting feature was evident in the SEM micrographs of the cycled plates (see Fig. 8a–c for uncompressed, 40 and 160 kPa compressed cells, respectively). The porous

material surrounding the corrosion layer demonstrated a significant increase in particle density as a function of compression pressure. This observation supports the theory that compression improves the agglomeration-of-spheres in the active material adjacent to the corrosion layer leading to improved connectivity of the active material, enhancing the performance of batteries. Furthermore, this is consistent with the EIS results, which showed that the reactivity of the CL + AMCL interface is enhanced considerably by the influence of plate compression.

4. Conclusions

With increased compression pressure, there is a remarkable increase in the cycle-life of lead-acid batteries. In terms of the EIS modelling, no trend could be seen between the cycle number and the shape of the complex-plane impedance plots, albeit that several tentative relationships existed between the equivalent circuit fitted parameters and compression pressure.

At full charge, all cells showed ideal capacitor behaviour and little difference existed between cells compressed at different pressures. At full discharge, however, the 160 kPa compressed cell demonstrated a much lower ohmic resistance for the CL + AMCL interface than the other cells, and a much higher reaction layer capacitance. The uncompressed cell and the cell compressed at 40 kPa tended towards a diffusion-controlled reaction at low frequency, while the electrochemistry in the cell compressed at 160 kPa was kinetically controlled. This behaviour symbolizes that the refinement of porosity of the positive plate at elevated compression enhances the diffusion rate of sulfuric acid into the porous AM causing a shift from a diffusion to activation controlled reaction chemistry.

SEM imaging showed a reduction in active material particle sizes and porosity in the vicinity of the corrosion layer with increased compression pressure, due to the enhanced connectivity of particles via the agglomeration-of-spheres, although the corrosion layer thickness seemed to increase as a function of compression.

Acknowledgements

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